



TITLE:

Analysis of the Mixture of Thiourea and Ammonium Thiocyanate

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thod as follows: 5~10 L of sea-water was taken, acidified with 20 ml of HCl and added 20 mg of Fe^{+++} as FeCl_3 solution. Then Fe^{+++} was precipitated with cupferon solution (5 g of cupferon), allowed to stand for 2~3 days. Then V was coprecipitated with Fe-cupferon nearly completely. The precipitate was filtered, ashed in platinum crucible and fused with small amounts of $\text{Na}_2\text{CO}_3 + \text{K}_2\text{CO}_3$. The fused mass was extracted with hot water. The solution was acidified with HNO_3 , added a drop of Br_2 water, and evaporated to dryness. The dried material was extracted with water, and the solution transferred to 10 ml measuring flask. Then added 1 ml of $\text{HNO}_3(1:1)$, H_3PO_4 (85%, 1:4) and Na_2WO_4 solution (0.2 M) respectively. Diluted to the mark and the resultant color of the solution measured with Purfrich-photometer by using filter S_{43} . In this V determination, $\times 40$ Co, $\times 40$ Ni, $\times 50$ Cu, $\times 20$ U, $\times 10$ Mo and < 3 mg Fe did not interfere.

Vanadium in the sea-water off shore of Shirahama, Wakayama Prefect., Japan was found to be 3~4 γ /L, and the mean value 3 γ /L.

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9. Analysis of the Mixture of Thiourea and Ammonium Thiocyanate

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On the analysis of the mixture of $\text{CS}(\text{NH}_2)_2$ and NH_4SCN studies have been made by Volhard, Reynolds & Werner, Storch, Krall, Gilfillan and Burrows¹⁾. As their methods determining $\text{CS}(\text{NH}_2)_2$ by iodometry and NH_4SCN by titration with AgNO_3 solution were found to be unsatisfactory, following method is proposed, which determines $\text{CS}(\text{NH}_2)_2$ by precipitation with xanthidrol-methanol solution and NH_4SCN by titration of the filtrate with AgNO_3 solution.

1. Analysis of a sample which contains only thiourea. The methanol solution of a sample is taken into a 500 cc. beaker, and a mixture of 2 % xanthidrol-methanol solution and glacial acetic acid, in which the former solution contains xanthidrol in the ratio 2~3 mol per 1 mol $\text{CS}(\text{NH}_2)_2$ in the sample solution and the quantity of the latter is 4 times in volume as much as that of the former solution, is added to the sample solution and agitated with a mechanical stirrer for 3 hrs. The beaker is then allowed to stand over night and the content is filtered through a glass filter. The precipitate is washed several times with methanol and water alternately, and in that case each of methanol and water is consumed 30 times in volume as much as the sample solution. The precipitate is dried for 1 hrs. at 110°C and is weighed

as xanthidryl-thiourea.

II. Analysis of a mixture of thiourea and ammonium thiocyanate.

Thiourea is determined by the exactly same procedure as described in 1.

For the determination of ammonium thiocyanate, 5 cc $\text{HNO}_3(1:7)$ and 5 drops of ferric alum solution are added to the filtrate obtained at the determination of thiourea and titrated with $\text{N}/10 \text{ AgNO}_3$ solution.

1) See ; Gilfillan, J. Am. Chem. Soc. 42, 2072 (1920) ;

Burrows, J. Am. Chem. Soc. 46, 1923 (1924).

10. Trial Construction of New Glass Capillary Viscosimeter

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There are various types of capillary viscosimeters but these viscosimeters have a defect that the maintenance of the constant pressure exerted in their capillary flow during each measurement is not easy. So the authors tried to construct a simple glass capillary viscosimeter in which the flow pressure is regulated automatically constant by a hydrostatic method. Employing this viscosimeter, the determination of viscosity of some pure organic solvents and 1 % aqueous solution of gelatin was carried out. The results thus obtained are given in the following tables.

Table 1. viscosity of pure organic solvents

sample	flow pressure P (dynes/cm ²)	flow time T (sec.)	P.T.	viscosity at 20°C (poise)
n-propanol	13370	63.0	842310	0.022799
	9750	86.4	842400	0.022702
	6400	131.6	842240	0.022697
nitro benzene	12310	60.8	748448	0.020131
	7020	106.6	748332	0.020127
aniline	22620	72.8	164673	0.04428
	16200	101.8	1649160	0.04435
	10850	151.6	1644860	0.04423

Table 2. viscosity of 1 % aqueous solution of gelatin

flow pressure P (dynes/cm ²)	flow time T (sec.)	P.T.	viscosity at 20°C (poise)
22246	10.0	222460	0.03816
8820	26.0	229320	0.04019
6370	93.0	592410	0.10939
3920	180.0	705600	0.12440